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SURVEY OF THE DEFORMATION CHARACTERISTICS OF TUNGSTEN

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INTRODUCTION

Tungsten, recognized today as one of the most promising structural materials for extreme high temperature applications, has not as yet reached its full potential due to the limitations imposed by its poor oxidation resistance and fabricability. Comparatively little work has been done until recent years to further its usefulness as a structural material and many investigations are now underway for this purpose.

Although tungsten has high strength near room temperature, its brittleness presents a problem, particularly in fabrication processes. Tungsten exhibits a ductile-brittle transition range above room temperature. This is characterized by a rapid increase in yield strength with decreasing temperature. When the stress required to produce yielding equals or exceeds the stress required to produce fracture, brittle failure occurs. A corresponding drop in ductility results down through the transition range. The temperature range denoting the ductile-brittle transition range is not fixed but is primarily dependent on the purity, microstructure, surface effects, and deformation rate, in addition to stress state. The effects of these variables will be considered for polycrystalline material and single crystals.

POLYCRYSTALLINE TUNGSTEN

The presence of impurities in tungsten generally increases the ductile-brittle transition temperature. Barth's[1] review illustrates an improvement in ductility may be achieved by vacuum annealing, instead of hydrogen annealing, tungsten. The data in Figure 1 shows the variation of reduction of area with temperature for both types of annealing treatments. The greater reduction of area for the vacuum-annealed specimens is credited to the lower impurity content produced by the vacuum anneal. Atkinson et al[2] compared the tensile properties of powder metallurgy tungsten of commercial and high purity and has shown that the high-purity material had a slightly lower transition temperature. Data for reduction of area for various test temperatures are shown in Figure 2. However, from the data obtained it was estimated that the tensile transition temperature of tungsten is not strongly influenced by differences in the concentrations of gaseous impurities when these are in the 10 to 50 weight parts per million range but may be affected by the presence of certain metallic impurities of the order of 10 to 30 weight parts per million. The speculative effect of trace metallic impurities has also been reported by Davis et al.[3] To date there have not been any correlations of mechanical properties with impurity type or magnitude.

Another phase of investigation to determine impurity effects was made by internal friction measurements.[2] These measurements were made in polycrystalline and single crystals of tungsten. Peaks were noted to occur in polycrystalline material, rather consistently in regions below room temperature, between 25 and 250 C, between 500 and 650 C, and at approximately 700 C. The peaks were produced by annealing and became more pronounced as the annealing temperature was increased. The peaks could not be precisely

identified with any particular impurity, however. The activation energies of the peaks as read from a Marx-Wert plot were given approximately as 20 to 30 kcal/mol for the 25 to 250 C peak region, 40 to 45 kcal/mol for the 500 C peak region, and 60 kcal/mol for the 700 C peak region. By comparison of activation energies derived from internal friction measurements with reported activation energies for diffusion, such as those compiled by Peterson,[4] identification of the various peaks may be possible. At this time it appears that more internal friction data and more reliable diffusion data would be required before any justified conclusions may be made with respect to specific causes or interpretations of the kinetics involved.

Data obtained by Bechtold and Shewmon[5] have shown a strain aging peak to occur at approximately 500 C. A strain rate of 2.8×10^{-4} inch/inch/second was used for testing. This peak was not identified with any specific impurity. According to theory, close similarity should be found between activation energies for diffusion and for strain aging. If speculations concerning internal friction-impurity diffusion relationships are presumed to be true, then one might consider carbon to be the impurity responsible for the strain aging at 500 C. This presumption is based on the fact that carbon has a higher solubility in tungsten than the other interstitials and is more difficult to reduce in low ppm levels. Also the reported activation energy for diffusion of carbon in tungsten approximates the activation energy determined for the internal friction peak occurring near 500 C.

The degree of impurity effect on the mechanical properties of tungsten is dependent on the microstructure of the particular material. The dependence of ductility on the microstructure has been effectively shown by various investigations.[6,7,8] Worked tungsten exhibits ductility at lower temperature levels than recrystallized material. Schwartzberg et al[7] illustrate that tungsten, reduced over 95%, exhibited ductility at room temperature. Curves illustrating this effect are shown in Figure 3, a plot of elongation versus temperature. Material worked 56% lost all ductility between room temperature and 100 C. Recrystallized tungsten showed a transition range between 100 C and 210 C. Microstructure resulting from various annealing temperatures were also shown to affect ductility.[6] Swaged powder metallurgy tungsten specimens were annealed at 1300, 1600, and 2100 C. The transition temperature was lowest for the specimens annealed at 1300 C as shown in Figure 4. The higher transition temperatures are apparently due to the greater degree of recrystallization occurring in the specimens annealed at the higher temperatures. The investigators evaluated the results in terms of substructural differences observed. The transmission electron microscopy work of Jones[8] also attributed loss of ductility after annealing to substructural changes. It seems apparent that primary and secondary recrystallization must be considered when variations in properties are attributed to microstructure.

Since recrystallization, or degree of recrystallization, plays an important role in influencing ductility of tungsten, investigations involving its study should be considered here.

Recrystallization studies, using pure and "doped" tungsten wire, were conducted by Rieck.[9] On recrystallizing, the texture found for the pure tungsten wire was the [110]. The doped wire consisted of both large and small crystals. The small crystals tended to keep the original deformation texture [110] while the large crystals have their [421] or [531] axis parallel to the wire axis. In rolled tungsten, however, annealing above 1800 C produced a texture {100} <011> $\pm 12^{\circ}$. Alternatively, this can be described as corresponding approximately to the orientations {011} <320> and {011} <230>. The as-rolled texture of the tungsten sheet was found to be centered about the orientation {100} <011>. It seems probable that the degree of anisotropy of certain crystal orientations in polycrystalline tungsten may have a significant effect on mechanical properties at the ductile-brittle temperature range as well as at high temperatures. The orientation effect will become more evident in the subsequent discussions on single crystal studies and in the studies of Peck and Thomas[10] on fibrous tungsten and iron. Fibrous microstructures produced in tungsten by drawing and swaging operations were studied by metallographic techniques and hardness determinations. Thin, curved grains were observed in the transverse sections of heavily drawn wires. The grain shape was attributed to the nonuniform mode of deformation of the grains associated with the characteristic [110] preferred orientation that develops in bcc metals during drawing. The percentage reductions used to vary the characteristic microstructure of the material were 39, 73, and 87%. It was estimated that thinning of the grains, or [110] texture development sufficient to produce thinning, begins at approximately 60% reduction. Above 70% reduction, it was noticed that the rate of work hardening increased.

This was explained by suggesting a change in relative degrees of slip occurring for various orientations. In other words, a higher degree of intersecting slip occurs at higher percentage reductions. The different orientations obtained for wire and sheet material were explained in terms of grain thinning, relative degrees of slip, and method of applied force. The lower ductile-brittle temperature for fibrous material (heavily worked material) was thought to occur as a result of the reduction of grain boundary area normal to the surface. It is suggested here that the specific orientation parallel to the longitudinal axis of the material is also significant.

An interesting observation made by Smithells[11] was cited where a tungsten bicrystal wire, with the boundary parallel to the axis, was shown to be ductile at room temperature. Although the specific crystal orientation was not mentioned, it is believed that the (110) was perpendicular to the wire axis. Therefore, being a cubic system, the [110] is assumedly parallel to the wire axis. From a subsequent discussion on single crystals, this direction appears most desirable for maximum ductility and minimum work hardening rate.

The ductility of tungsten has been shown to be profoundly influenced by the deformation rate.[12, 13] The effect of this variable may be evidenced at any temperature range. Increasing the rate of strain results in a

corresponding decrease in ductility at or near transition zone temperatures. However, at elevated temperatures (>3000 F), an increase in the rate of strain increases the ductility. Effects of temperature and strain rate on the ductility of wrought tungsten were investigated by Magnussen and Baldwin. Reduction of area values for two different strain rates for various temperatures are shown in Figure 5. The data shown here appears to be quite scattered, however. Further tensile transition results were also reported for recrystallized powder metallurgy tungsten tested at strain rates of 0.008, 0.8, and 80 inches per minute.[6] The data is shown graphically in Figure 6. For a particular temperature, the extent of decreased ductility with increasing strain rates can be readily seen. A plot of the reciprocal of the transition temperature versus log strain rates (Figure 7) for all of these results shows direct proportionality between the data, as mentioned above, for the wrought and recrystallized tungsten.

As mentioned previously, increasing the strain rate at elevated temperatures increases the ductility. An investigation conducted by Sikora and Hall,[13] using strain rates of 0.002, 0.2, and 20 inch/inch/minute, showed that the increase in reduction of area at 3500 F was 15%, 58%, and 99% respectively. They also found that increasing the strain rate from 0.002 to 20 inch/inch/minute approximately quadrupled the ultimate strength at 4000 F. Fractures were generally transgranular at the higher strain rates and intergranular at the lower strain rates. The results obtained for the various investigations on strain rate effects suggest that at intermediate temperatures, an increasing strain rate alters the direction of reduction of area values from a decreasing trend to an increasing one.

The effect of surface condition on the properties of tungsten specimens has been observed by Stephens,[14] Atkinson et al,[2] and Steigerwald and Guarnieri.[15] Decreasing ductility has been associated with an increase in surface irregularities, imperfections, and/or the formation of a dislocation barrier at the surface to cause dislocation pile-up from the interior and from Stroh cracks. It has been determined that the removal of a surface layer by electropolishing or oxidation on machined specimens can restore ductility. (This is shown in Figure 8.) Rescratching the electropolished specimens with fine emery paper resulted in decreased ductility whereas the same procedure applied to the oxidized specimens showed no decrease in ductility. It was assumed that oxidation provided a scratch barrier for protection during subsequent handling. The improvements in transition behavior could not be quantitatively correlated to the surface profile. The frequency and acuity of the surface defects was considered to be significant.

SINGLE CRYSTALS

The study of single crystals has usually involved the use of high-purity specimens. However, in some investigations, the single crystal was intentionally dosed with impurities to determine their effect in the absence of grain boundaries. An investigation of this type was conducted by Allen, Maykuth and Jaffee.[16] Impurities of oxygen, carbon, and nitrogen were intentionally added to single crystals of tungsten. Tensile tests

at room temperature showed that all of the dosed crystals retained good ductility (over 50% reduction of area). The crystals dosed with oxygen showed the greatest reduction in ductility from that of the pure tungsten crystals. Some crystals were subjected to tensile tests at 400 C. X-ray analysis of the fracture areas indicated that the introduction of carbon, oxygen, or nitrogen did not change the mode of deformation. Solid solubility limits for interstitial contaminants in tungsten were roughly determined as follows: carbon - 100 to 200 ppm, oxygen - 30 to 40 ppm, and nitrogen - 1 to 2 ppm. Solubilities were determined for temperatures of 2000 C, 1700 C, and 2300 C respectively. The solubility values are in approximate agreement with those of Klopp and Barth.[17]

The room temperature tensile tests showed that crystals containing up to 36 ppm of carbon or 39 ppm of oxygen showed good to excellent ductility.

The increase in transition temperature of polycrystalline material over that of single crystals was proposed to result from surface notch effects of the high angle grain boundaries.

An investigation on single crystals of tungsten was also conducted by Ferris, Rose, and Wulff.[18] This study was directed toward a determination of orientation effects in single crystals. The three basic orientations used here were the [110], [111], and [100]. Crystals with the various axial orientations were tested at temperatures of 77 K, 195 K, 300 K, and 373 K. Strain rates varied from 2.5 inch/inch/minute to 2.5×10^{-4} inch/inch/minute. The activation energy for yielding of single crystals, at 300 K and a strain rate of 0.025 inch/inch/minute, was found to be insensitive to crystal orientation, and had a value of approximately one-half electron volt. The activation volume was found to be sensitive to crystal orientation for the same temperature and strain rate of 300 K and 0.025 inch/inch/minute. The quantities were determined to be $7b^3$ for [110] crystals, $12b^3$ for [100] crystals, and $14b^3$ for [111] crystals. The proportional limit was also found to be dependent on orientation. To show appreciable deviation from proportionality, the stress required for the [110] crystals was 3 times greater than that required for the [100] crystals. A [111] crystal required between 2 and 2.5 times the stress required for the [100] crystal. The [110] crystals exhibited a yield phenomenon and a low degree of work hardening. The [111] and [100] crystals yielded smoothly and work hardened to a high degree. A possible explanation for the yielding variation with orientation was based on the fact that a <110> type tensile specimen can form only one set of <100> dislocations as a result of slip. However, both the [111] and [100] specimens can form at least three nonparallel sets. If cross-slip in tungsten occurs readily, then the [110] oriented crystals should show the least amount of work hardening. In addition, the single set of [100] dislocations which could form for [110] tensile specimens would have Burgers vectors perpendicular to the tensile axis. The <111> and <100> type tensile specimens are capable of forming [100] dislocations with components parallel to the tensile axis. Only in the latter case should these [100] dislocations nucleate fracture as proposed by Cottrell.[19]

One might compare the results of this investigation with those obtained by other studies on the orientation dependence of deformation characteristics in alpha-iron.[20,21] In general, the data appears to be somewhat analogous. If we assume a similarity in characteristics, it would be interesting to determine whether observations made with tungsten single crystals could be generally complemented by the following assumptions:

1. Crystals with a [100] orientation exhibit a greater degree of twinning than either the [110] or [111] crystals.

2. Increasing the strain rate or decreasing the temperature increases the degree of twinning.

3. Prestraining at some specific temperature (>room temperature) should reduce or eliminate the tendency to twin at lower temperatures.

From the results of Biggs and Pratt[20] on alpha-iron at -183 C, the ductile-brittle range occurring between the [110], [111], and [100] orientations could be extended further toward the [100] orientation by decarburizing or the addition of 0.3% manganese (Figure 9). Cleavage fracture was postulated to be nucleated by the pile-up of dislocations against the boundary of a twin, which has already been nucleated by the burst of dislocations released at the upper yield stress.

An investigation of the temperature dependence of the yield strength of tungsten single crystals was conducted by Schadler and Low.[22] Tests were performed at temperatures of 20, 77, and 298 K at a strain rate of 2 percent per minute. Electropolishing or electromachining was used for final processing of the specimens. Crystals of varied orientations were used for the tests. From the data obtained on crystals deformed at 77 and 20 K, the active slip plane was determined to be the (011) and the twinning plane is the (112). At 298 K, deformation occurred by slip on the (011) and the (112) planes. Room temperature fracture was reported to occur by chisel-type fracture or by cleavage on (001) type planes. Although no correlation was given for the orientation dependence of chisel and cleavage failures at room temperature, the results may be indicative of those obtained by Ferriss, Rose and Wulff.[18] The stress at the proportional limit, determined at temperatures of 20, 77, and 298 K, was resolved as shear stress on the (011) planes and in the [111] direction. The resolved shear stress was plotted as a function of reciprocal temperature.

It was mentioned that using the resolved shear stress on the (112) [111] systems where (112) slip was observed did not alter the average value of the resolved shear stress within the limits of the standard deviation. Although the method of testing the single crystals was somewhat different than that used by Ferriss et al, the strain rates used to determine stress value at the proportional limit were approximately the same. However, the orientation dependence of the stress value at the proportional limit found by one investigation is not reflected in the reported results of the other. Upon further testing of single crystal specimens[23] with consideration to

orientation effects, presented data was more analogous to that of Ferriss et al. Figure 10 shows relative data for the temperature dependence of the stress at the proportional limit.

In a study conducted to investigate twinning in single crystals of tungsten,[24] crystals were grown with a reported purity of 99.99%. Some crystals were rolled at temperatures of 400 C and 1100 C. Some were deformed in compression at room temperature, while others were swaged in air at 700 C to 1500 C. After deformation, extensive twinning was observed. Analysis showed the twin plane to be {112}. Twinning direction was presumed to be <111>. Several general types of twins were evident. Some were long lamellar twins with irregular sides, while others had a serrated side. Some twins contained microcracks. The microcracks appeared to initiate within the twin band. They are presumed to be initiated by the twinning action. After swaging a specimen from a preheat temperature range of 700 C to 1500 C, twins were evident on the fracture surface. It was apparent that twinning inhibits recrystallization in single crystals of tungsten. This was demonstrated by deforming single crystals at various temperatures and then recrystallizing in vacuum. The lower deformation temperature caused the highest recrystallization temperature. The ability to twin appeared to be dependent on the number of refining passes or residual impurities. Crystals having fewer passes twinned more readily than those subjected to a higher number of passes.

Twinning studies were also conducted by Probst.[25] Twins were produced in tungsten single crystals by explosive working at room temperature. Although the explosive shock fractured the crystals into fragments of various size, these were sufficiently large to be used for the study. All crystals exhibited profuse twinning. Most of the twins had jagged twin boundaries and were found to be parallel to {112} planes. The irregular twin boundaries are grooved surfaces in which the sides of the grooves appear to be parallel to {110} planes and the grooves themselves are parallel to a <111> direction. It might be mentioned here that the grooved twins may be equivalent to some of those found by Schnitzel and Keith[24] in their single crystal deformation studies.

An investigation on the fabrication and properties of tungsten and tungsten alloy single crystals was performed by the staff of Linde and Haynes Stellite Company.[26] Major effects on fabrication and properties of tungsten single crystals were attributed to chemical composition, orientation, and low angle boundaries. After the crystals were prepared, working was accomplished by one of two methods. The first method consisted of centerless grinding followed by a pickling in a 60-40 HNO_3-HF solution. After this treatment, the crystals were swaged. The second method was to pickle in the 60-40 HNO_3-HF solution, then forge to a flat plate, pickle again, and then roll. A temperature of approximately 1200 C was used for most of the fabricating.

The swaged specimens were tested in tension at 1000 C and showed good mechanical properties. The hot-rolled crystals, which were reduced to

0.040" thick sheet, were subjected to bend tests to determine ductile-brittle transition temperatures. Although the [100] axially oriented material appeared to have the lowest ductile-brittle transition temperature, more data would be required for confirmation of these results.

The presence of grain boundaries, produced by working the single crystals, made fabrication at lower temperatures virtually impossible. Certain alloying additions, such as Zr, Hf, and Ir, were found to cause severe embrittlement, probably due to the formation of a second phase.

It was determined that [100] oriented material exhibited the best working characteristics. This result appears to be in agreement with other reported data on orientation effects if one considers that the [110] direction, for this case, is favorably oriented in the sheet for deformation by rolling. The [110] direction is reported as exhibiting the minimum degree of work hardening in single crystals.

Single crystals of tungsten were tested by Atkinson et al[2] to determine the various effects of impurities, orientation, etc., on the properties of the crystals. Ground tensile specimens exhibited brittle cleavage with very little plastic deformation. All crystals were made ductile, however, by electropolishing the surface prior to testing. This is attributed to complete removal of the heavily worked surface.

It was discovered that by annealing a single crystal in hydrogen, a distinct yield point could be observed when the specimen was strained at -62 C. The upper yield load and the flow stress were greatly increased.

Very limited orientation studies showed that chisel fractures always occurred for specimens tested at 300 K. However, the orientation of all these specimens was close to the [110]. This is in agreement with other reported data.

CONCLUSIONS

From the evaluation of reported data, it is apparent that much more work must be accomplished in order to quantitatively correlate the effects of the variables discussed with the mechanical properties of tungsten. However, from the reported data of many investigations, some significant correlations and evaluations may be made. Although they may be more qualitative than quantitative, the speculations, based on available data, may provide a basis for direction of future studies.

Results of basic investigations have shown that variable purity, microstructure, surface condition, and deformation rate will significantly affect the mechanical properties of tungsten, particularly near the ductile-brittle transition temperature range.

From the standpoint of purity, oxygen and/or carbon appear to be particularly detrimental as interstitial contaminants. The effect of trace

metallic impurities is unknown but is suspected to produce a significant effect in property variations. Thus far, the inability to correlate a specific impurity effect with a property variation has been due to the incapabilities of present analytical techniques at low impurity levels. Internal friction techniques are not without promise and they are expected to yield informative data on impurity effects in tungsten.

The effect of surface condition on the mechanical properties has been already demonstrated. Both electropolishing and oxidation have a beneficial influence. Further data should be accumulated for effects of surface conditions with respect to orientation, percent reduction by deformation, and perhaps the effect of various types of diffusion layers into the surface. More data is required on mechanical property variations with a change in deformation rate. Specific correlations are in order for change in orientation, microstructure, and effect with specific impurities. One example of the latter effect would be the apparent increase in temperature at which strain aging occurs with an increased deformation rate. One may consider whether the same interstitial is responsible for both peaks or whether the change in deformation rate has produced effectiveness in a different interstitial.

In summation, it may be stated that much more quantitative information of a basic nature is required before a comprehensive evaluation can be made on the particular characteristics of tungsten. It is apparent that a number of factors will significantly influence mechanical property measurements and all should be taken into consideration for accurate data analysis.

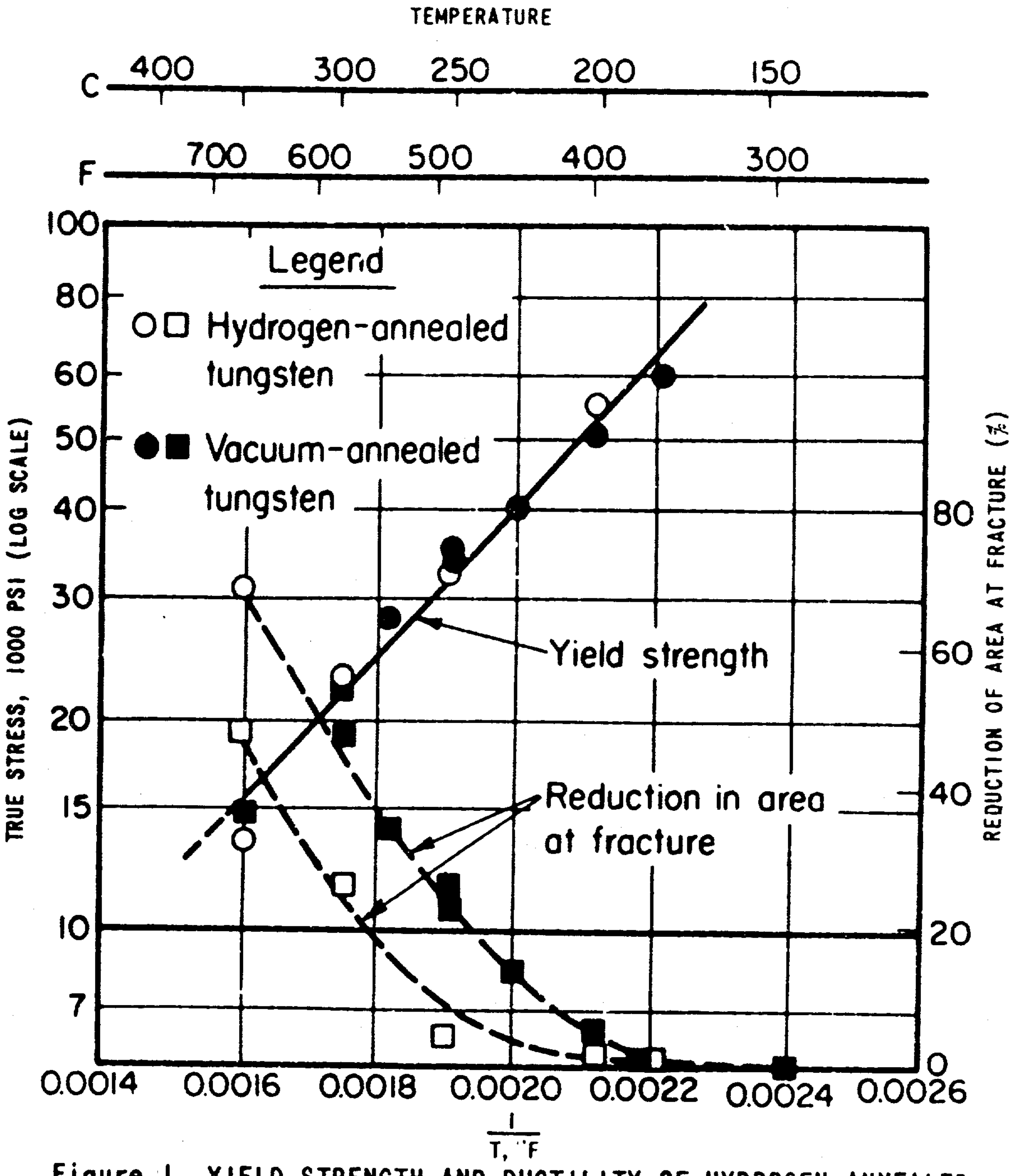

Figure 1. YIELD STRENGTH AND DUCTILITY OF HYDROGEN-ANNEALED AND VACUUM-ANNEALED TUNGSTEN [12]

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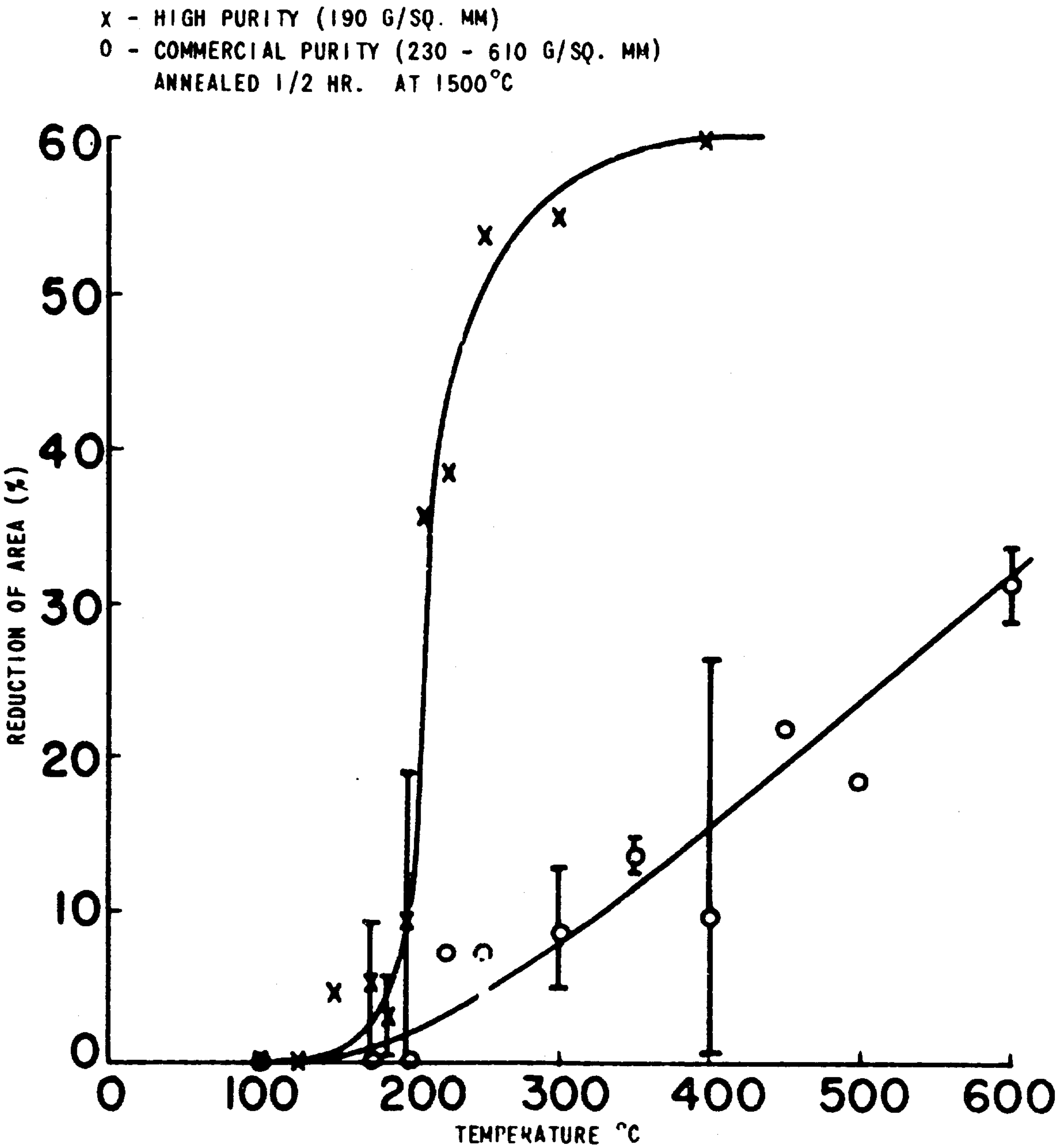

Figure 2. REDUCTION OF AREA VERSUS TEST TEMPERATURE (80% COLD WORK)[2]

19-366-900/AMC-63

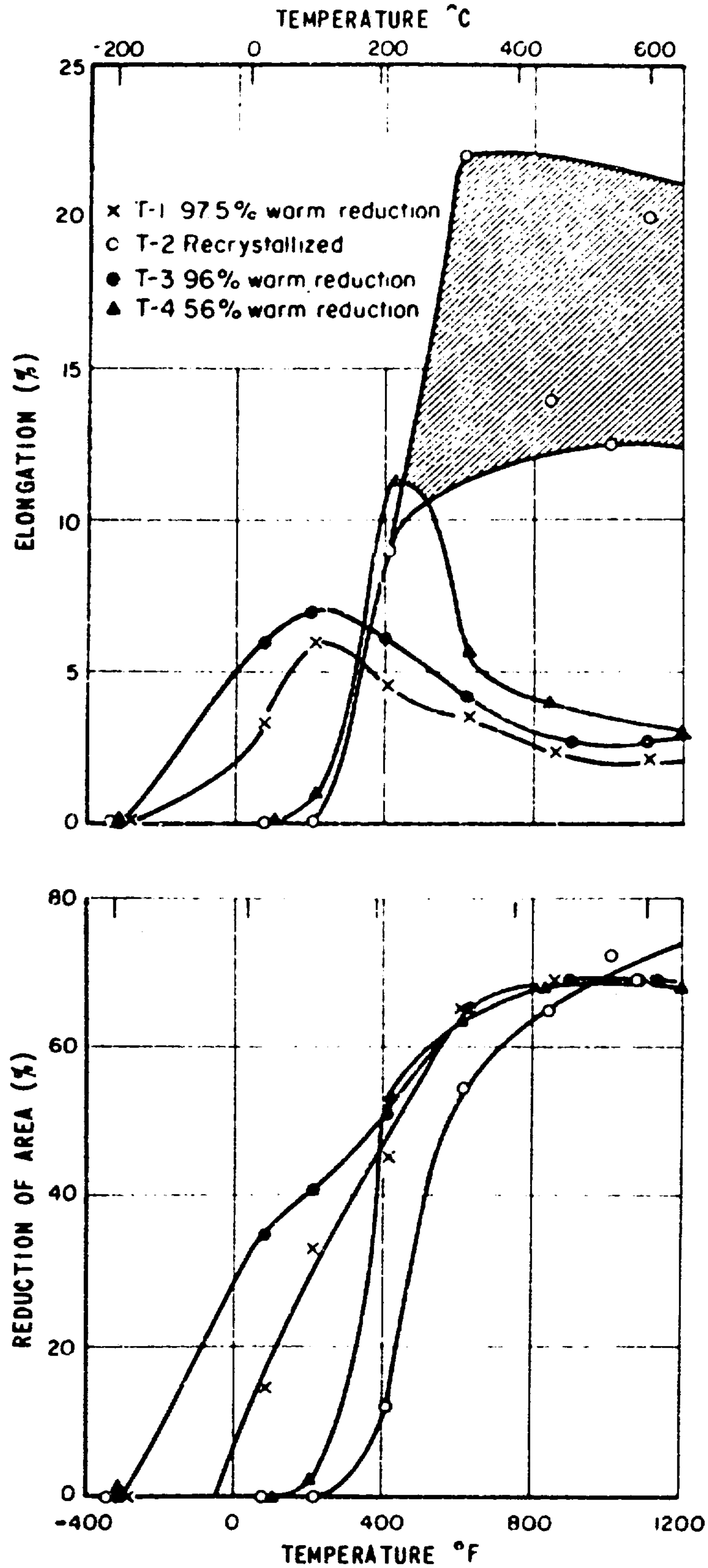

Figure 3. EFFECT OF TEMPERATURE ON THE TENSILE DUCTILITY OF TUNGSTEN WIRE[27]

19-066-899/AMC-63

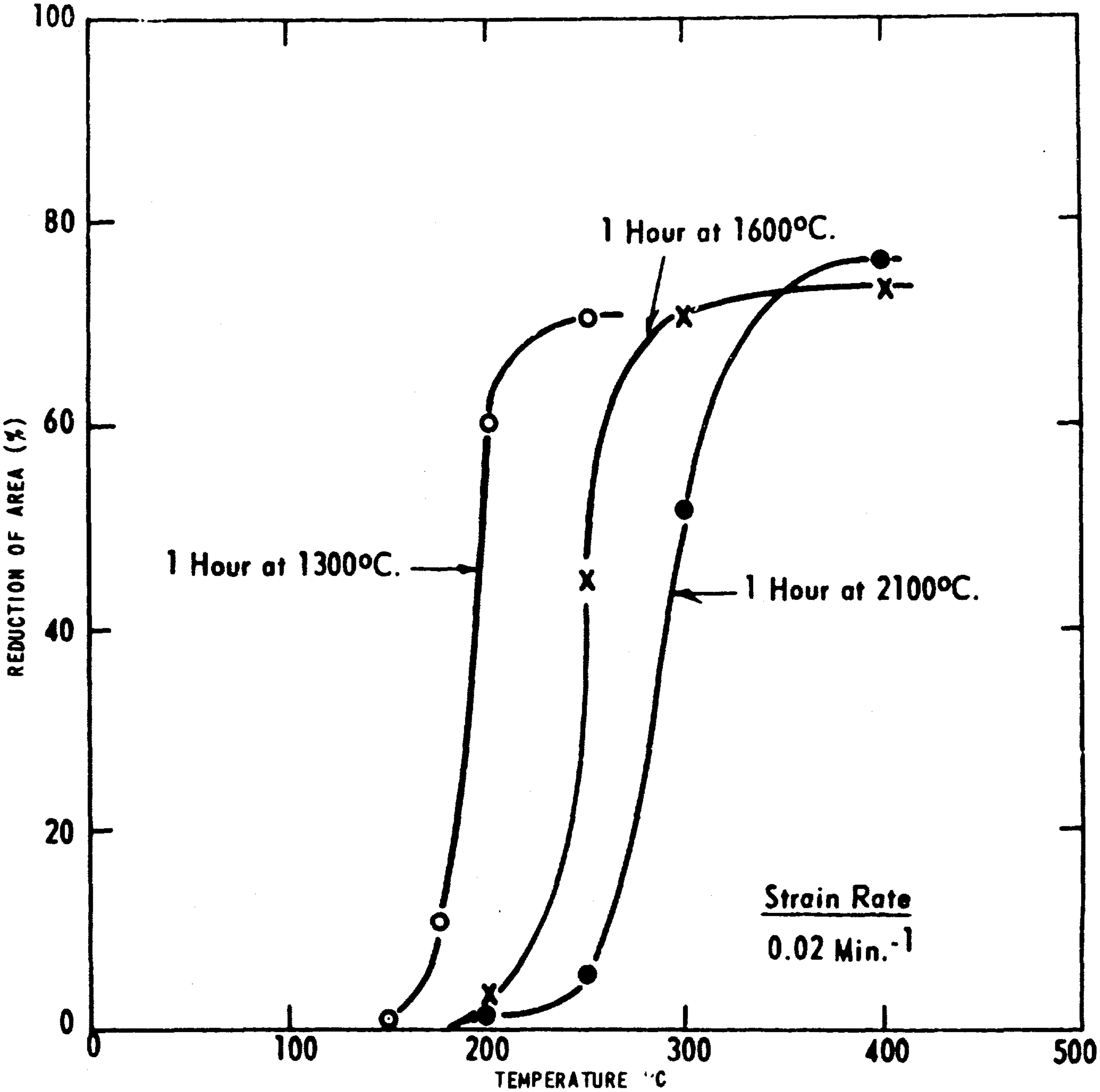

Figure 4. EFFECT OF ANNEALING TEMPERATURE ON THE DUCTILE-BRITTLE TRANSITION TEMPERATURE OF SWAGED POWDER METALLURGY TUNGSTEN[6]

19-665 898/AMC-6

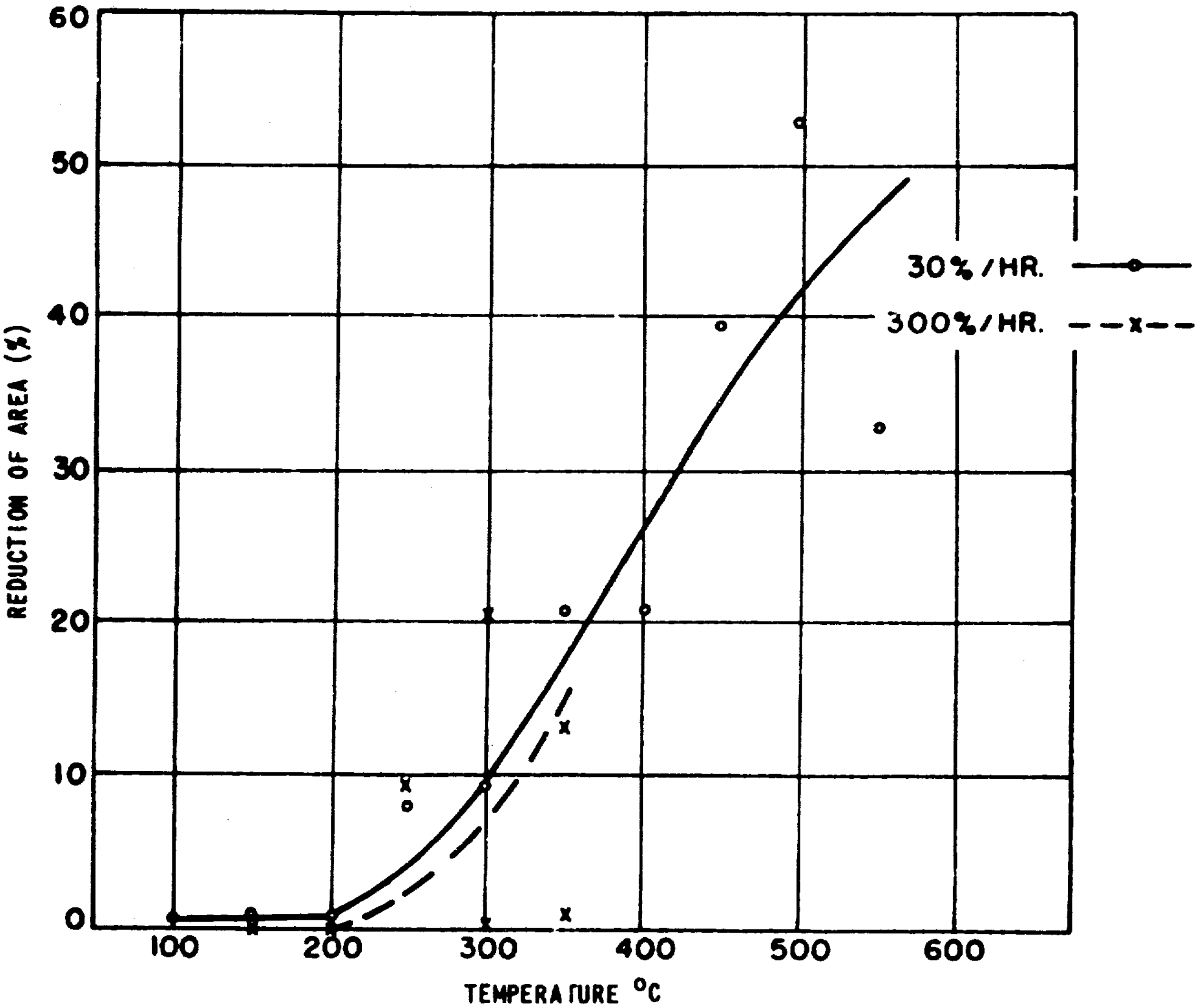

Figure 5. EFFECT OF STRAIN RATE ON THE TRANSITION TEMPERATURE OF COMMERCIAL PURITY TUNGSTEN[2]

19-066-901/AMC-63

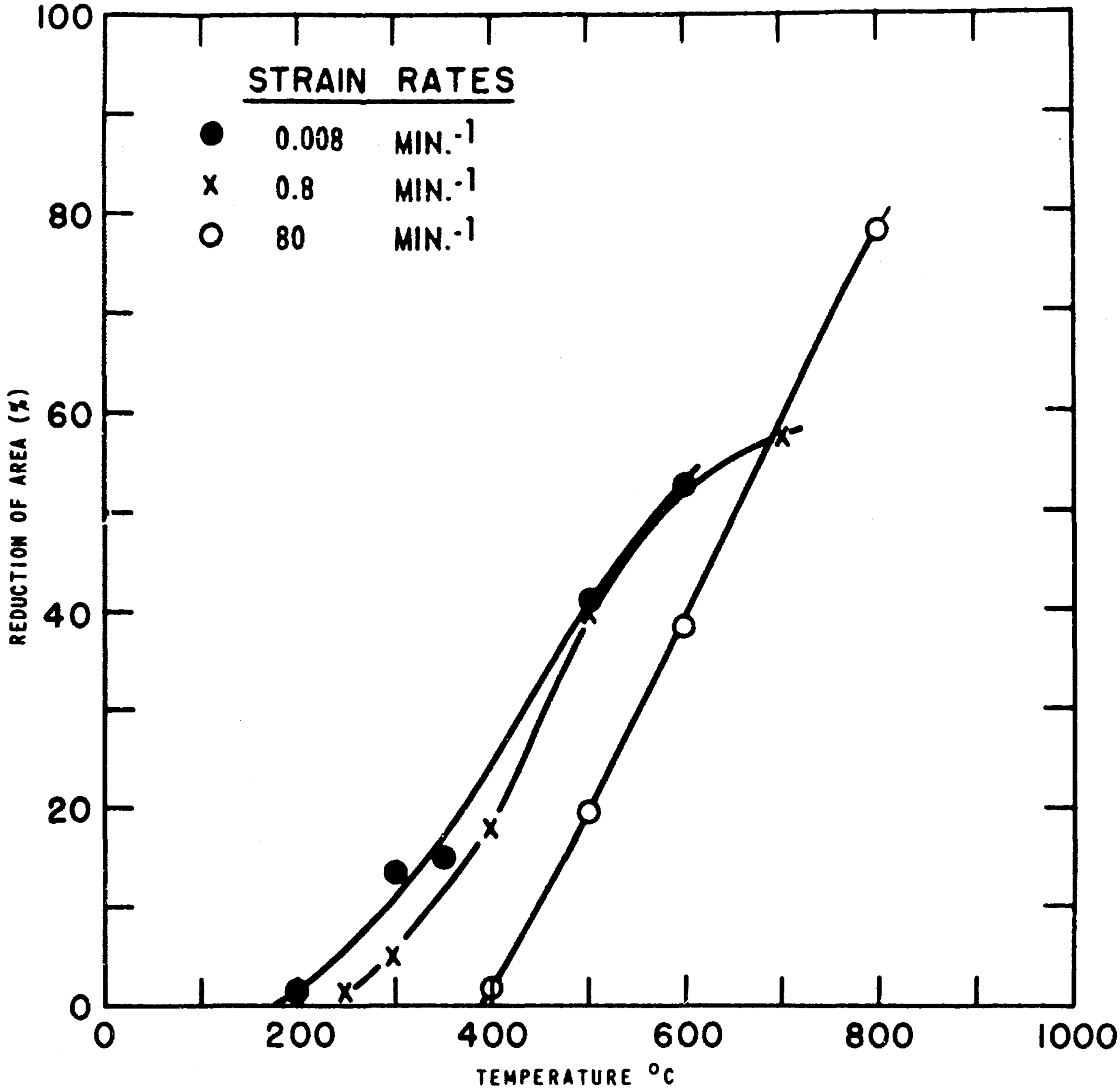

Figure 6. REDUCTION OF AREA VERSUS TEMPERATURE FOR RECRYSTALLIZED TUNGSTEN TESTED AT VARIOUS STRAIN RATES[6]

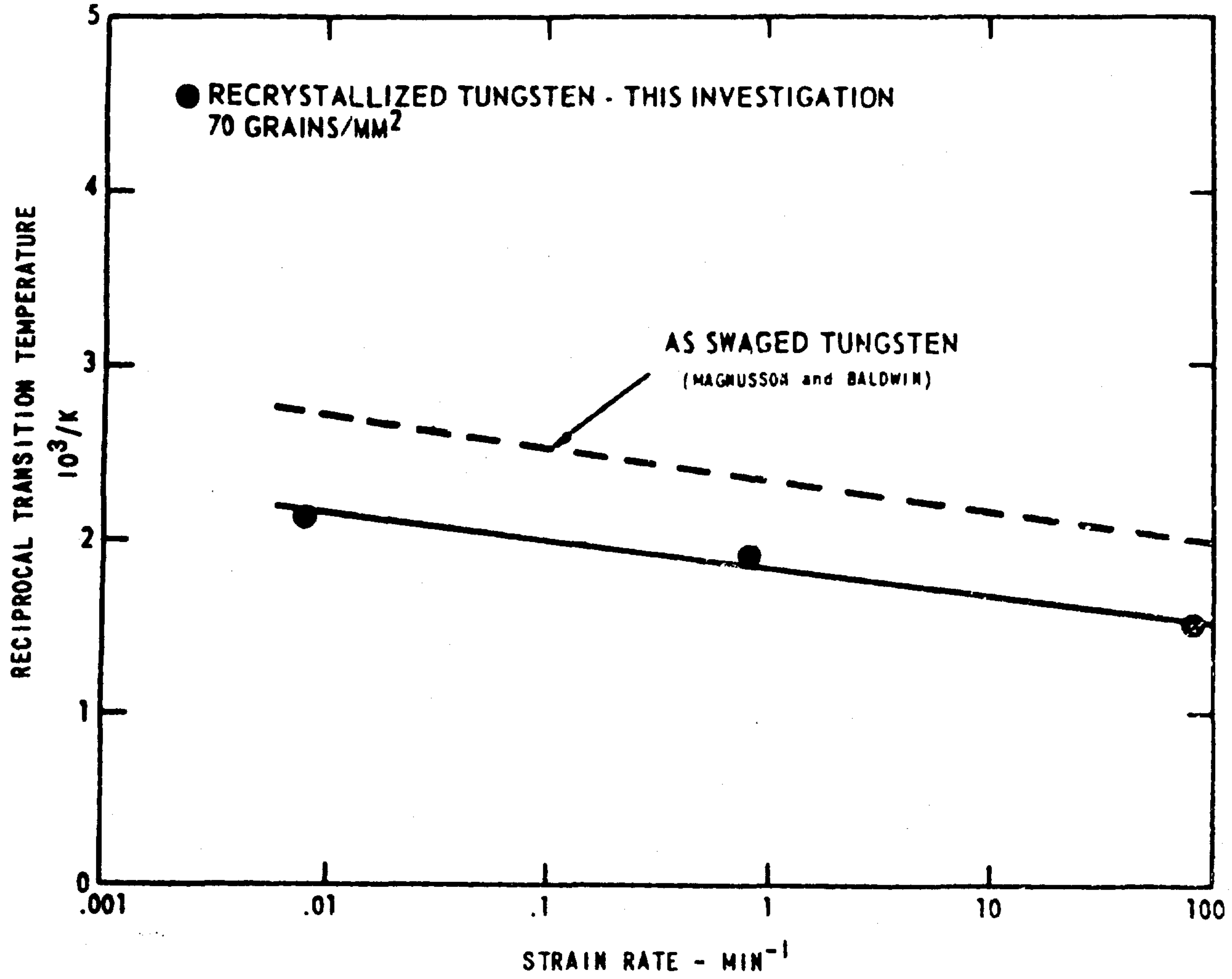

Figure 7. STRAIN RATE DEPENDENCE OF DUCTILE-BRITTLE TRANSITION TEMPERATURE OF TUNGSTEN[6]

19-066-896/AMC-63

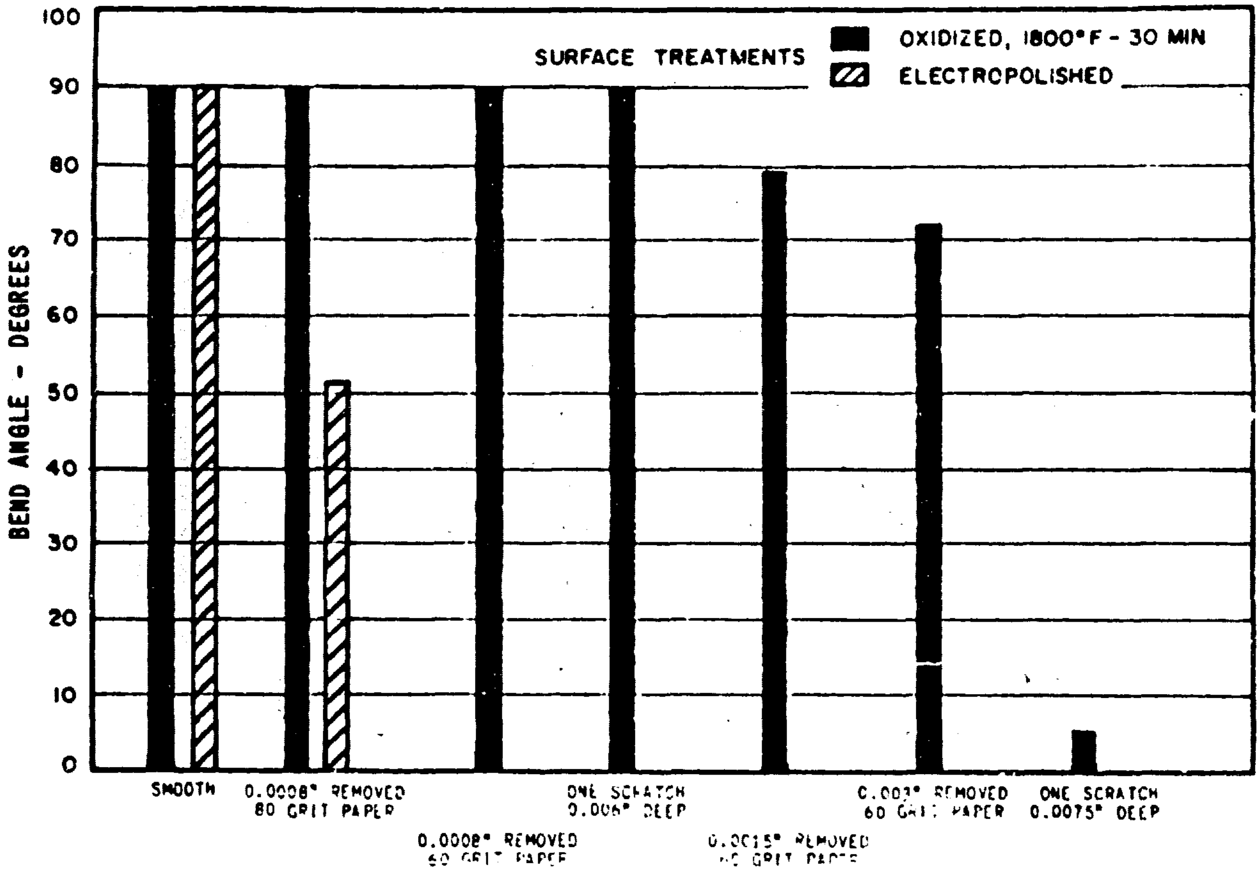

Figure 8. INFLUENCE OF SURFACE IMPERFECTIONS APPLIED AFTER SURFACE TREATMENT ON THE BEND ANGLE OF TUNGSTEN SPECIMENS TESTED AT 380°F[15]

19-066-895/AMC-63

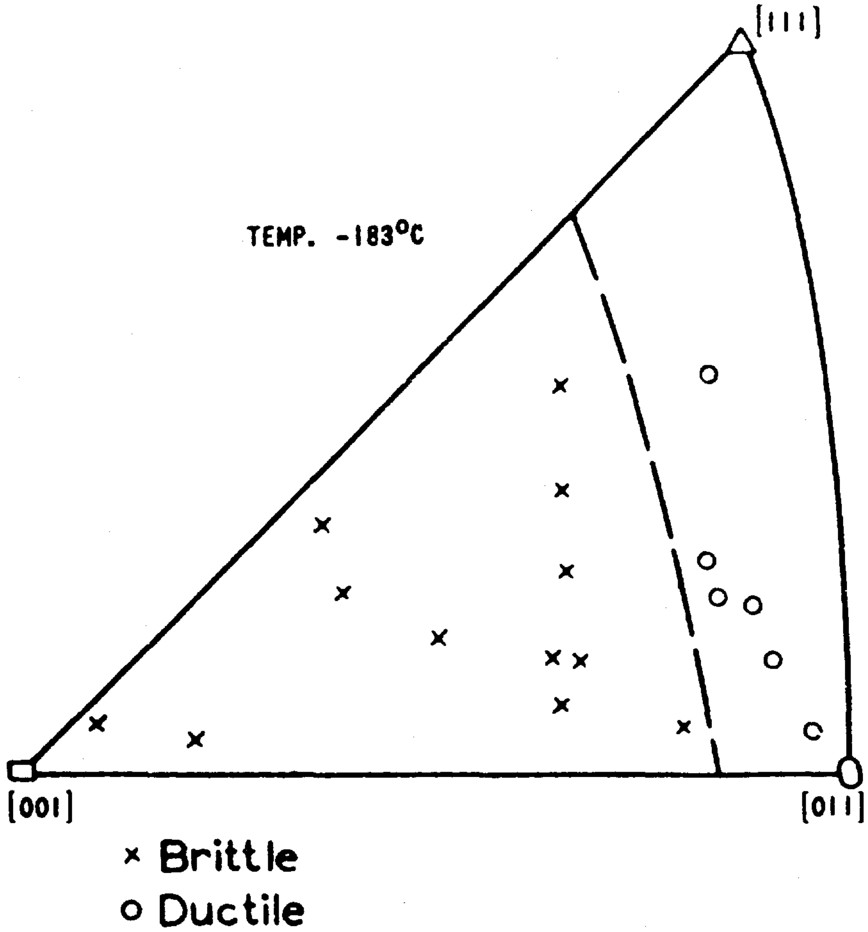

Figure 9. SHIFT OF DUCTILE - BRITTLE TRANSITION WITH RESPECT TO ORIENTATION FOR ALPHA-IRON[20]

19-066-894/AMC-63

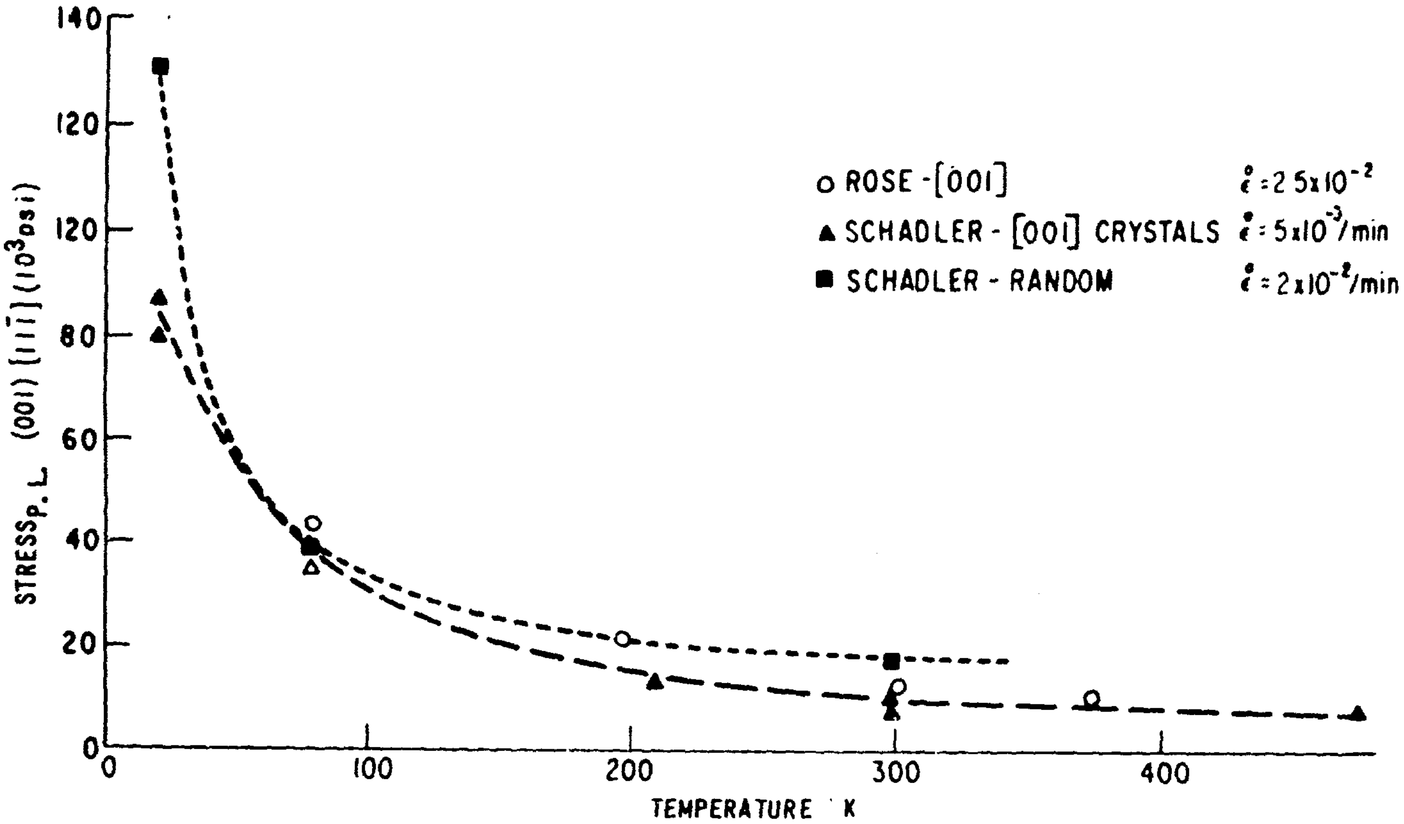

Figure 10. TEMPERATURE DEPENDENCE OF THE PROPORTIONAL LIMIT OF [001] AXIS AND RANDOM AXIS SINGLE CRYSTALS[23]

19-066-893/AMC 63

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